

# PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2000-006345

(43)Date of publication of application : 11.01.2000

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(51)Int.Cl. B32B 27/36

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(21)Application number : 10-172735 (71)Applicant : MITSUBISHI POLYESTER FILM  
COPP

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## (54) COMPOSITE POLYETHYLENE NAPHTHALATE FILM

### (57)Abstract:

PROBLEM TO BE SOLVED: To obtain a film, which is excellent in transparency and thermic rays cutting properties and can obtain an excellent energy saving effect under the condition being used over a long term by pasting to the window of an automobile or the like by including metal-added oxidized metal particles or the like in a resin layer formed on one side of a laminated film consisting of polyethylene naphthalate layers A, B and C and a dye and the like in the layer B.

SOLUTION: Polyethylene naphthalate is a polymer substantially consisting of ethylene-2,6-naphthalate unit as its building block and coextruded in a film and then biaxially stretched so as to laminate three layers A, B and C to one another. In a resin layer formed on one side of the film, metal-added oxidized metal particles or oxidized metal particles such as titanium dioxide particles, calcium carbonate particles or the like are included. As the dye to be included in the layer B, a natural dye such as indigo or the like and a synthetic dye such as an azo dye, an anthraquinone dye or the like. As the pigment, an organic pigment such as a phthalocyanine-based dye or the like and an inorganic pigment such as titanium white, zinc white, carbon black or the like are exemplified.

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### LEGAL STATUS

[Date of request for examination] 09.05.2005

[Date of sending the examiner's decision of

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**DETAILED DESCRIPTION**

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**[Detailed Description of the Invention]****[0001]**

[Field of the Invention] This invention relates to a coloring polyethylenenaphthalate film with good heat ray cut nature in detail about a laminating polyethylenenaphthalate film. Conventionally, as film which is penetrable to the light of a visible region and has reflexivity or absorptivity to the light of an infrared region, the coat of indium oxide and tin oxide is prepared by sputtering on a biaxial-stretching polyethylene terephthalate film, and forming a selection light transmission nature layered product is indicated (JP,57-67441,A etc.). On the other hand, the constituent or coating film which blended the organic system infrared absorption agent in addition to metallic oxides, such as antimony dope tin oxide, indium dope tin oxide, and a vanadium oxide, is indicated by JP,9-310031,A, JP,9-316115,A, and JP,9-316363,A.

[0002] However, in order to obtain the selection light transmission nature layered product which prepared the coat of indium oxide and tin oxide by sputtering on the polyethylene terephthalate film, equipment [ need / the ambient atmosphere where a high vacuum and precision are high / to be controlled ] must be used, and the magnitude of that a production cost becomes high and the film which can be created has a limit. On the other hand, the coating film by the constituent which blended the organic system infrared absorption agent in addition to metallic oxides, such as antimony dope tin oxide, indium dope tin oxide, and a vanadium oxide, has the low permeability of a light field, and it is hard to come out of the original color of the colored film when a film is colored. Moreover, in order that an organic system infrared absorption agent may absorb infrared radiation, it is in a difficult situation for degradation of an organic system infrared absorption agent to tend to take place, and to maintain fixed infrared absorption capacity for a long period of time.

[0003] Moreover, the film colored by the high-pressure staining technique etc. absorbs a beam of light, when sunrays are irradiated, and it is in the situation that a coloured film tends to deteriorate. Thus, the colored film maintains the original color for a long period of time, there is a limitation in suppressing degradation of the tenebrescence by sunrays and a film, and amelioration is called for.

**[0004]**

[Problem(s) to be Solved by the Invention] This invention is made in view of the above-mentioned actual condition, the solution technical problem has good transparency, and it is in offering the film which the clear nature when seeing an image through a film was secured, and was excellent in heat ray cut nature, and was excellent in energy saving, scattering prevention of glass, etc. when it was used to the aperture of vehicles, such as an aperture of an automobile, an aperture of an electric car, and an aperture of an airplane, the aperture of a building, etc., having been stuck for a long period of time.

**[0005]**

[Means for Solving the Problem] According to [ as a result of repeating examination wholeheartedly in view of the above-mentioned technical problem ] the laminated film of a specific configuration, this invention persons find out that the above-mentioned technical problem is easily solvable, and came to complete this invention. That is, the summary of this invention consists in the compound-ized

polyethylenenaphthalate film characterized by having a resin layer in one field of the film with which the laminating of at least three polyethylenenaphthalate layers, an A horizon, a B horizon, and C layer, was carried out one by one, for the resin layer concerned containing a metal addition metal oxide particle or a metal oxide particle, and said B horizon containing a color or a pigment.

[0006]

[Embodiment of the Invention] Hereafter, this invention is further explained to a detail. Although the polyethylenenaphthalate said by this invention points out the polymer by which the configuration unit is substantially constituted from ethylene -2 and a 6-naphthalate unit, small quantity, for example, less than [ 10 mol % ], the ethylene -2 which denaturalized by the third component not more than 5 mol %, and 6-naphthalate polymer are also contained preferably.

[0007] Polyethylenenaphthalate is manufactured by generally carrying out condensation of naphthalene -2, 6-dicarboxylic acid or its functional derivative -2, for example, naphthalene, and 6-dicarboxylic acid dimethyl and ethylene glycol to the bottom of a suitable reaction condition under existence of a catalyst. In this case, divalent alcohol, such as hydroxy acid like dicarboxylic acid, such as an adipic acid, a sebacic acid, a phthalic acid, isophthalic acid, a terephthalic acid, naphthalene -2, and 7-dicarboxylic acid, or the low-grade alkyl ester of those, and a p-oxy-benzoic acid, its low-grade alkyl ester or propylene glycol, a trimethylene glycol, tetramethylene glycol, pentamethylene glycol, and hexamethylene glycol, etc. can be mentioned as the third component.

[0008] If the polyethylenenaphthalate used by this invention has too low polymerization degree, since a mechanical property will fall, the limiting viscosity has the desirable thing of 0.45-0.9 to 0.40 or more and a pan. In this invention, although it does not matter even if a particle contains and polyethylenenaphthalate does not carry out, when it contains, thermally stable polymer pulverized coal which is indicated by a titanium dioxide, a calcium carbonate, magnesium oxide, a silicon dioxide, a kaolin, talc, a zeolite, lithium fluoride, a barium sulfate, carbon black, and JP,59-5216,B is mentioned as a particle which can be used. These particles are used independently and also they may use two or more sorts together.

[0009] In this invention, especially as an approach of making polyethylenenaphthalate containing a particle, it is not limited and a well-known approach can be adopted. For example, although it can add in the phase of arbitration which manufactures polyethylenenaphthalate, it may add as a slurry which ethylene glycol etc. was made to distribute preferably in the phase after the phase of esterification, or ester exchange reaction termination, and before polycondensation reaction initiation, and a polycondensation reaction may be advanced. Moreover, it is carried out by the approach of blending the dried particle and a polyethylenenaphthalate raw material etc. using the approach of blending the slurry and polyethylenenaphthalate raw material of the particle which ethylene glycol or water was made to distribute, or a kneading extruder using a kneading extruder with a vent.

[0010] In this invention, although not limited, the range of 0.05 micrometers - 1 micrometer especially of 0.05 micrometers - 0.7 micrometers of mean particle diameter of the particle which a polyethylenenaphthalate film contains is 0.05-0.5 micrometers still more preferably preferably. When mean particle diameter is less than 0.05 micrometers, a particle condenses and it is easy to become a big and rough particle. Moreover, when mean particle diameter exceeds 1 micrometer, the surface roughness of a film becomes large too much, and a film may become hazy.

[0011] the laminating polyethylenenaphthalate film said to this invention -- all layers -- extrusion -- it is the film by which melting push appearance is carried out from both mouthpieces and which was extruded by the so-called co-extruding method, and is the film made [ 2 shaft orientations of a lengthwise direction and a longitudinal direction ] to carry out orientation to behind. The particle may be contained in the A horizon and C layer which constitute the laminating polyethylenenaphthalate film of this invention. As a particle to contain, particles, such as thermally stable polymer pulverized coal which is indicated by a titanium dioxide, a calcium carbonate, magnesium oxide, a silicon dioxide, a kaolin, talc, a zeolite, lithium fluoride, a barium sulfate, carbon black, and JP,59-5216,B, are mentioned, for example. These particles may use together independent or two sorts or more.

[0012] In this invention, especially as an approach of blending the above-mentioned particle with

polyethylenenaphthalate, it is not limited and a well-known approach can be adopted. For example, although it can add in the phase of arbitration which manufactures polyethylenenaphthalate, it may add as a slurry which ethylene glycol etc. was made to distribute preferably in the phase of esterification, or the phase before after [ ester exchange reaction termination ] polycondensation reaction initiation, and a polycondensation reaction may be advanced. Moreover, it is carried out by the approach of blending the dried particle and a polyethylenenaphthalate raw material etc. using the approach of blending the slurry and polyethylenenaphthalate raw material of the particle which ethylene glycol or water was made to distribute, or a kneading extruder using a kneading extruder with a vent.

[0013] Although not limited, the range of 0.05-1 micrometer especially of 0.05-0.7 micrometers of mean particle diameter of the particle which the A horizon and C layer which constitute the laminating polyethylenenaphthalate film of this invention contain is 0.05-0.5 micrometers still more preferably preferably. When mean particle diameter is less than 0.05 micrometers, a particle may condense and it may become a big and rough particle. Moreover, when mean particle diameter exceeds 1 micrometer, the surface roughness of a film becomes large too much, and a film may become hazy.

[0014] The B horizon which turns into an interlayer of the laminating polyethylenenaphthalate film of this invention contains a color or a pigment. It is required for an interlayer to contain a color or a pigment in respect of tenebrescence prevention. In this invention, especially as an approach of blending a color and a pigment into poly ETEREN naphthalate, although not limited, it can blend by the well-known approach. That is, a staining technique with well-known approach of carrying out the dryblend of a color or a pigment, and the polyethylenenaphthalate raw material, and extruding them, high-pressure staining technique, etc. is mentioned using a kneading extruder.

[0015] As a color, it can classify into natural dye and synthetic dye, and indigo (indigo) etc. is represented as natural dye. As synthetic dye, azo dye, anthraquinone dye, indigoid dye, sulfur dye, triphenylmethane dye, pyrazolone dye, stilbene dye, diphenylmethane dye, xanthene dye, alizarin dye, acridine dye, a quinonimine dye (for example, azine dye, an oxazine color, thiazine dye), thiazole dye, methine dye, nitro dye, nitroso dye, cyanine dye, etc. are mentioned.

[0016] As a pigment, it can classify into an organic pigment and an inorganic pigment, and Quinacridone, watch ANGU red, dioxazine violet, etc. are mentioned as a typical thing with pigments, such as a phthalocyanine system, a dioxazine system, and an anthraquinone system, as an organic pigment. Moreover, as an inorganic pigment, a titanium white, a zinc white, the white lead, carbon black, red ocher, a vermillion, cadmium red, the chrome yellow, ultramarine blue, cobalt blue, cobalt purple, Zinc chromate, etc. are mentioned.

[0017] these colors and pigments -- one sort -- or two or more sorts can be used, using together. The content of the color in the interlayer B horizon which constitutes the film of this invention, and a pigment is 0.01 - 10 % of the weight usually 0.1 - 5% of the weight of the range still more preferably 0.05 to 7.5% of the weight preferably. There is an inclination for there to be few degrees of coloring and for heat ray cut nature to fall [ this content ] at less than 0.01 % of the weight. Moreover, even if it makes it contain exceeding 10 % of the weight, the degree of coloring is already saturated.

[0018] Although not limited, it can be made to contain by the well-known approach in this invention especially as an approach of making a color and a pigment containing in poly ETEREN naphthalate. That is, the approach that the approach of carrying out the dryblend of a color or a pigment, and the polyethylenenaphthalate raw material, and extruding them etc. is well-known is mentioned using a kneading extruder.

[0019] Next, although the manufacture approach of the laminating polyethylenenaphthalate film in this invention is explained concretely, the laminating polyethylenenaphthalate film of this invention is not limited to the following examples of manufacture at all. That is, the polyethylenenaphthalate raw material described previously is used, using two or more sets of extruders, the multi-manifold die of two or more layers, or a feed block, the laminating of each poly ETEREN naphthalate is carried out, the melting sheet of two or more layers is usually extruded and carried out at 290-330 degrees C from a mouthpiece, cooling solidification is usually carried out with a 40-80-degree C cooling roller, and a non-extended sheet is obtained. In this case, in order to raise the smoothness of a sheet, it is desirable to raise

the adhesion of a sheet and a rotation cooling drum, and electrostatic impression contact printing and/or liquid spreading contact printing are adopted preferably. In this invention, both may be used together if needed.

[0020] Thus, biaxial stretching of the obtained amorphism sheet is carried out to serial or coincidence, and it is heat-treated at about 180-270 degrees C after that so that it may become length to a degree and it may usually become about 4 to 20 times for an area scale factor in a longitudinal direction by 130-170 degrees C. It faces extending in length and a longitudinal direction, and you may extend respectively in one step, and if needed, it can extend multistage or the heat treatment section for orientation relaxation can also be prepared between multistage extensions. Furthermore, after biaxial stretching, before presenting the heat treatment process of degree process, you may extend again. Especially for high-intensity-izing, after performing 1.05 to 4.0 times as many re-extension as this in length and a longitudinal direction at the temperature of further 140-200 degrees C after biaxial stretching, the approach of heat-treating is good.

[0021] The so-called in-line coating which processes a film front face in an extension process can be performed. Although it is not limited to below, the first step of extension is completed, and before the second step of extension, they are the purposes, such as amelioration of antistatic nature, slipping nature, an adhesive property, etc., and fabricating nature amelioration, and can perform coating processing of a water solution, a drainage system emulsion, a drainage system slurry, etc., for example.

[0022] That what is necessary is just the thickness which can produce a film as a film, preferably, when the laminating polyethylenenaphthalate film of this invention is used as the film of 10-50-micrometer thickness still more preferably, it can demonstrate 10-100 micrometers of 5-200 micrometers of outstanding effectiveness. Moreover, as for the rate of the thickness of the A horizon and C layer which constitute a laminating polyethylenenaphthalate film, and the B horizon which is an interlayer, it is desirable that it is the range of 90 / 10 - 10/90.

[0023] The resin layer by which a laminating is carried out to at least one side of the laminated film of this invention contains a metal addition metal oxide particle or a metal oxide particle. As a metal addition metal oxide particle or a metal oxide particle A tin addition indium oxide particle (ITO), a tin oxide addition indium oxide particle, A stibiation tin oxide particle (ATO), a stibiation zinc-oxide particle (AZO), A cadmium addition tin oxide particle (CTO), a magnesium addition indium oxide particle (MgIn<sub>2</sub>O<sub>4</sub>), A cadmium addition oxidation gallium particle (CdGa<sub>2</sub>O<sub>4</sub>), a zincing oxidation gallium particle (ZnGa<sub>2</sub>O<sub>4</sub>), Although an indium addition tin oxide particle (In<sub>4</sub>Sn<sub>3</sub>O<sub>12</sub>), indium oxide addition tin oxide, a gallium addition indium oxide particle (GaInO<sub>3</sub>), a zincing indium oxide particle (ZnIn<sub>2</sub>O<sub>4</sub>), tin oxide, indium oxide, a zinc oxide, etc. are mentioned The metal added by the metal addition metal oxide particle may be an oxide, and may be plural. These particles may be contained independently or two or more kinds may be contained in coincidence.

[0024] Also in these, a tin addition indium oxide particle (ITO), a stibiation tin oxide particle (ATO), an indium oxide particle, and a tin oxide particle are desirable, and especially a tin addition indium oxide particle (ITO) is desirable in respect of transparency and heat ray cut nature. Moreover, even if these particles are calcinated, and not calcinated, they are not cared about. Although the ambient atmosphere of the baking may be in a non-oxygen condition (for example, an anoxia condition and reduced condition) or you may be in an owner oxygen condition when calcinated, what was calcinated in the state of non-oxygen from the point of the heat ray cut engine performance is desirable.

[0025] in addition, the configuration of these particles -- the shape of massive, a globular shape, and an ellipsoid, and needlelike \*\* -- it is good anything. The mean particle diameter of the metal addition metal oxide particle used in this invention and a metal oxide particle has 0.2 micrometers or less and desirable 0.1 more micrometers or less. When mean particle diameter exceeds 0.2 micrometers, there is an inclination for the transparency over a visible ray to fall.

[0026] The resin used for the resin layer by which a laminating is carried out to at least one side of the film of this invention Polyester resin, acrylic resin, polyurethane resin, polycarbonate resin, Silicone resin, an epoxy resin, polystyrene resin, ABS plastics, polyvinyl chloride resin, Polyvinylidene chloride resin, polyolefin resin, polyamide resin, water-soluble alkyd resin, It can be used combining one sort or

two kinds or more from resin, such as polyvinyl alcohol resin, poly butyl alcohol resin, polyvinyl butyral resin, polyvinyl-acetal resin, vinyl acetate resin, and acrylic-styrene resin.

[0027] In this invention, it is desirable to adopt an activity energy-line hardening resin layer as a resin layer, and especially as an ingredient which constitutes an activity energy-line hardening resin layer, although not limited, various kinds of cross-linking resin is mentioned, for example. As an example of cross-linking resin, the resin of acrylic, a polyester system, an urethane system, a melamine system, an epoxy system, and an organic silicate system, the copolymer resin of a \*\* silicon compound and a fluorine-containing compound, etc. are mentioned. In these, activity energy-line hardening resin is desirable in respect of productivity etc.

[0028] As activity energy-line hardening resin, hardening components, such as an unsaturated-polyester-resin system, acrylic, an addition polymerization system, a hybrid system of a thiol acrylic, a cationic polymerization system, and a hybrid system of cationic polymerization and a radical polymerization, can be used. Also in these, hardening resin acrylic at points, such as hardenability, abrasion-proof nature, surface hardness, flexibility, and endurance, is desirable.

[0029] The above-mentioned acrylic hardening resin contains the acrylic oligomer and the reactant diluent as an activity energy-line polymerization component. And a photopolymerization initiator, a photopolymerization initiation assistant, a modifier, etc. are contained if needed. Typically as acrylic oligomer, the oligomer by which a reactant acryloyl radical or a reactant meta-acryloyl radical was combined with the acrylic resin frame is mentioned. As other acrylic oligomer, polyester (meta) acrylate, epoxy (meta) acrylate, urethane (meta) acrylate, polyether (meta) acrylate, silicone (meta) acrylate, polybutadiene (meta) acrylate, etc. are mentioned. Furthermore, the oligomer which the acryloyl radical or the meta-acryloyl radical combined with the frame with upright melamine, isocyanuric acid, annular phosphazene, etc. is mentioned.

[0030] Since itself has the acrylic oligomer of polyfunctional or monofunctional nature, and the radical which reacts while bearing the function of the solvent in a spreading process as a medium of the paint, a reactant diluent serves as a copolymerization component of a paint film. As an example of a reactant diluent, pen TAERISURITORUTORI (meta) acrylate, Pentaerythritol tetrapod (meta) acrylate, JIPENTAERISURITORUTORI (meta) acrylate, Dipentaerythritol tetrapod (meta) acrylate, dipentaerythritol PENTA (meta) acrylate, Dipentaerythritol hexa (meta) acrylate, TORIMECHI roll pro pantry (meta) acrylate, Ethylene glycol (meta) acrylate, propylene GURIKORUJI (meta) acrylate, acryloyloxypropyl (meta) triethoxysilane, acryloyloxypropyl (meta) trimethoxysilane, etc. are mentioned.

[0031] as a photopolymerization initiator -- a 2 and 2-ethoxy acetophenone, 1-hydroxy cyclohexyl phenyl ketone, dibenzoyl, a benzoin, benzoin methyl ether, benzoin ethyl ether, benzoin iso-propyl ether, p-chlorobenzo phenon, p-methoxybenzophenone, a Michler's ketone, an acetophenone, 2-chloro thioxan ton, anthraquinone, phenyl disulfide, and 2 - methyl-[4-(methylthio) phenyl]-2-morpholino-1-propanone etc. is mentioned.

[0032] As a photopolymerization initiation assistant, thioether systems, such as alkylphosphine systems, such as tertiary amine systems, such as triethylamine, triethanolamine, and 2-dimethylamino ethanol, and triphenyl phosphine, and beta-thiodiglycol, etc. are mentioned. As a modifier, a spreading nature amelioration agent, a defoaming agent, a thickener, an inorganic system particle, an organic system particle, lubricant, an organic macromolecule, a color, a pigment, a stabilizer, etc. are mentioned. These are used in the range which does not check the reaction by the activity energy line, and can improve the property of an activity energy-line hardening resin layer according to an application. An organic solvent can be blended with the constituent of an activity energy-line hardening resin layer for control of the improvement in workability at the time of coating, and coating thickness.

[0033] He is philharmonic polyethylenenaphthalate to whom biaxial orientation of the laminating approach of an activity energy-line hardening resin layer was carried out in this invention. Although the approach of making one side of MU apply and harden a hardenability resin constituent, and carrying out a laminating is illustrated, it is not limited to this laminating approach at all. In this invention, it can coat as a concrete approach of applying a hardenability resin constituent by well-known approaches, such as

the reverse roll coat method, the gravure roll coat method, the rod coat method, and the Ayr knife coat method.

[0034] With heat etc., after the applied hardenability resin constituent dries a solvent, it can be stiffened with an activity energy line and can form a hardening coat. In that case, as an activity energy line, it is desirable to form a hardening coat according to ultraviolet rays, a visible ray, an electron ray, an X-ray, alpha rays, beta rays, a gamma ray, etc. When irradiating an activity energy line and making a hardening coat form, although an exposure is performed from a spreading layer side, in order to raise adhesion with a film, you may carry out from the opposite side side of a spreading layer, and the reflecting plate which may reflect an activity energy line further may usually be formed. The coat hardened especially by the activity energy line has well desirable abrasion resistance.

[0035] In this invention, 0.5-10 micrometers is usually the range of 1-5 micrometers preferably as thickness of an activity energy-line hardening resin layer. When abrasion resistance may fall when the thickness of a hardening resin layer is less than 0.5 micrometers, and exceeding 10 micrometers, hardening contraction of a wear-resistant layer is large, and a film may curl to a wear-resistant layer side.

[0036] In this invention, although how to carry out the laminating of the resin layer which contains a metal addition metal oxide particle and a metal oxide particle in one field of a polyethylenenaphthalate film is explained concretely, it is not limited to the following approaches. Namely, the resin containing polyethylenenaphthalate, a metal addition metal oxide particle, or a metal oxide particle is extruded to coincidence using two or more extruders. Make it join by the inside of a feed block, or opening Kaneuchi, and carry out an extrusion laminating. The resin containing the so-called co-extruding method, a metal addition metal oxide particle, or a metal oxide particle is fused with an extruder. The so-called knockout laminating method which carries out a direct laminating on a polyethylenenaphthalate film, Apply on a polyethylenenaphthalate film as a slurry which contains a metal addition gold oxide group particle or a gold oxide group particle to the resin which dissolved in water or alcoholic solvent after casting of film production Rhine of a polyethylenenaphthalate film, or uniaxial stretching. the so-called in-line coating (ILC) -- the so-called off-line coating (OLC) applied to the resin which dissolved in the organic solvent on law and a polyethylenenaphthalate film as a coating containing a metal addition metal oxide particle or oxidation metal particles -- law etc. is mentioned.

[0037] Next, although the manufacture approach which carries out the laminating of the resin layer to the polyethylenenaphthalate film of this invention with an off-line coating method is explained concretely, the laminated film of this invention is not limited to the following examples of manufacture at all. That is, the coating which made the resin dissolved in the organic solvent distribute a metal addition metal oxide particle or oxidation metal particles can be created, and it applies on a polyethylenenaphthalate film by the method of application of a conventional method, it can be made to be able to dry and solidify, and a laminated film can be obtained. In addition, in coatings, additives, such as a dispersant and a coupling agent, may be added idiomatically.

[0038]

[Example] Hereafter, although an example explains this invention to a detail further, this invention is not limited to the following examples, unless the summary is exceeded. In addition, it shows the "weight section" that it is with the "section" among an example and the example of a comparison. Moreover, the measuring method used by this invention is as follows.

[0039] (1) Mean particle diameter (d<sub>50</sub>)

centrifugation type particle-size-distribution measuring device SA-CP by Shimadzu [ Corp. ] Corp. 3 mold -- using -- the resistance rule of a stokes -- a basis -- the magnitude of a particle was measured with the \*\*\*\* sedimentation method.

(2) The place separated from the heat ray cut nature sample film (300mmx300mm) 50mm was made to turn on the incandescent lamp of 100W, the back of a hand was held up to the place separated from the sample film 50mm to an incandescent lamp and the opposite side immediately after lighting and after 30 minutes, and the following rank estimated heat ray cut nature.

[0040]

## [Table 1]

----- rank A: Hardly feel a heat ray (level which is satisfactory practically).

Rank B: A heat ray is sensed a little (level which is satisfactory practically).

Rank C: A heat ray is sensed strong (level which has a problem practically).

----- [0041] (3) Light was irradiated on the sample film for 1000 hours using the long life type fading-test machine by Suga Test Instruments [ Co., Ltd. ] Co., Ltd. whenever [ color change ], the visual judgment of the color difference in trial order was carried out, and the rank was attached as follows.

[0042]

## [Table 2]

----- rank A: There is almost no difference (level which is satisfactory practically).

Rank B: There is a difference a little (level which is satisfactory practically).

Rank C: There is a difference (level which has a problem practically).

----- [0043] (4) On the gradation color scale in a clear nature GE plan pin center, large laser dot color chart [1], it separated 10cm, the sample film was placed in parallel, the visual judgment of the clear nature was carried out from the sample film side, and it divided into the following ranks.

[0044]

## [Table 3]

The rank D hard to see to 10% location of rank C: friend point concentration where 5% location of -----

----- rank A: rank [ which does not almost have a difference compared with a subject copy ]

B: friend point concentration is hard to see a little: ----- hard to see to 20% location of friend point concentration [0045] Here, Ranks A and B are level which is satisfactory practically.

<Manufacture of polyethylenenaphthalate> Example 1 (polyethylenenaphthalate A) of manufacture While carrying out the heating temperature up of naphthalene -2, the 6-dicarboxylic acid dimethyl 100 section, the ethylene glycol 60 section, and the magnesium acetate 4 monohydrate 0.09 section for the reactor, the methanol was distilled off, the ester exchange reaction was performed, 4 hours was required from reaction initiation, the temperature up was carried out to 230 degrees C, and the ester interchange was terminated substantially. Subsequently, the silica particle 0.07 section of 1.54 micrometers of mean diameters was added as an ethylene glycol slurry, the phosphoric-acid 0.03 section and the antimony-trioxide 0.035 section were added further, the polycondensation reaction was performed with the conventional method, and the polyethylenenaphthalate of limiting viscosity 0.50 and 0.07 % of the weight of silica particle contents was obtained. Solid state polymerization of the obtained polymer was carried out at 0.3mmHg(s) and 240 degrees C for 8 hours, and the polyethylenenaphthalate A of limiting viscosity 0.65 was obtained.

[0046] The example 2 (polyethylenenaphthalate B) of manufacture

In the example 1 of manufacture, Polyethylenenaphthalate B was obtained like the example 1 of manufacture except having not added the ethylene glycol slurry containing the 0.07 sections of silica particles of 1.54 micrometers of mean diameters to the system of reaction.

[0047] The example 3 (polyethylenenaphthalate C) of manufacture

the polyethylenenaphthalate B manufactured in the example 2 of manufacture -- drying -- the PORIE ethylene naphthalate B100 section -- receiving -- the Mitsubishi Chemical Diaresin yellow F -- the 0.4 sections and this Diaresin red HS -- the 0.7 sections -- said -- kneading extrusion and the coloring polyethylenenaphthalate C were obtained for Diaresin blue H3G with the 0.8 \*\*\*\*\* twin screw extruder.

[0048] The example 4 (polyethylenenaphthalate D) of manufacture

the polyethylene terephthalate B manufactured in the example 2 of manufacture -- drying -- the polyethylenenaphthalate B100 section -- receiving -- the Mitsubishi Chemical Diaresin yellow F -- the 0.8 sections and this Diaresin red HS -- the 1.4 sections -- said -- kneading extrusion and the coloring polyethylenenaphthalate D were obtained for Diaresin blue H3G with the 1.6 \*\*\*\*\* twin screw extruder.

[0049] The example 5 (manufacture of ITO powder) of manufacture

The mixed water solution of a nitric-acid indium and sulfuric-acid tin (mole ratio = 9 to 1) is received in a surfactant (twin 20 by the KISHIDA chemistry company) at a mixed water solution. After 1-% of the weight addition, After adding ammonia and carrying out coprecipitation of a hydroxylation indium and the hydroxylation tin, the coprecipitate was heated at 500 degrees C among the oxidizing atmosphere, 250 degrees C of obtained powder were further heat-treated with the rotary kiln reactor of a carbon monoxide gas ambient atmosphere for 60 minutes, deoxidation processing was performed in part, and yellow-green ITO powder was obtained.

[0050] The example 6 (coating liquid A) of manufacture.

The coating liquid A which prepared acrylic resin, the solvent (a xylene / MEK=80/20), and the surfactant to the ITO powder obtained in the example 5 of manufacture, and was made to distribute by the sand mill for 24 hours, and the ITO particle with a mean particle diameter of 0.05 micrometers contained 65% of the weight was obtained.

The example 7 (coating liquid B) of manufacture

In the example 6 of manufacture, coating liquid B was obtained like the example 6 of manufacture except not containing ITO powder.

The example 8 (coating liquid C) of manufacture

In the example 6 of manufacture, coating liquid C was obtained like the example 6 of manufacture except containing the titanium oxide particle of 0.27 micrometers of mean diameters instead of ITO powder.

[0051] Each chip of example 1 polyethylenenaphthalate A and C was dried in the 4-hour inert gas ambient atmosphere at 180 degrees C, respectively, appearance unification was carried out, cooling solidification was carried out on the cooling roller which becomes three layers of A/C/A within a feed block about melting extrusion and these melting polymers at 290 degrees C with the melting extruder according to individual and which set skin temperature as 40 degrees C using electrostatic impression contact printing, and the non-extended sheet was obtained. The obtained sheet was extended to the lengthwise direction 3.6 times at 130 degrees C. Subsequently, after leading the film to the tenter and extending in a longitudinal direction 3.6 times at 135 degrees C, heat setting was performed at 240 degrees C, and the coloring laminated film which is 25 micrometers whose lamination is A/C/A, and whose thickness of each class is 10/5/10 (micrometer) was obtained. The coat was carried out to one side of the obtained film by MEIYABA so that the thickness after desiccation might be set to 1.8 micrometers in coating liquid A, and it dried at 100 degrees C, and the compound-ized polyethylenenaphthalate film was obtained.

[0052] In example 2 example 1, the compound-ized polyethylenenaphthalate film was obtained like the example 1 except having carried out the coat of the coating liquid A so that the thickness after desiccation might be set to 0.8 micrometers.

[0053] In example 3 example 1, the compound-ized polyethylenenaphthalate film was obtained like the example 1 except having carried out the coat of the coating liquid A so that the thickness after desiccation might be set to 2.7 micrometers.

In example 4 example 1, the compound-ized polyethylenenaphthalate film was obtained like the example 1 except changing Polyethylenenaphthalate C into Polyethylenenaphthalate D.

[0054] In example of comparison 1 example 1, the compound-ized polyethylenenaphthalate film was obtained like the example 1 except having used coating liquid B instead of coating liquid A.

In example of comparison 2 example 4, the compound-ized polyethylenenaphthalate film was obtained like the example 4 except having used coating liquid B instead of coating liquid A.

[0055] In example of comparison 3 example 1, the compound-ized polyethylenenaphthalate film was obtained like the example 1 except having used coating liquid C instead of coating liquid A. As mentioned above, the obtained result is collectively shown in the following table 1.

[0056]

[Table 4]

表1

	実 施 例				比 較 例		
	1	2	3	4	1	2	3
熱線カット性	A	A	A	A	D	C	A
色変化度	A	A	A	A	B	C	A
鮮明性	A	A	A	A	A	A	D
総合評価	良好	良好	良好	良好	不良	不良	不良

[0057]

[Effect of the Invention] The effectiveness which was excellent in energy saving, scattering prevention of glass, etc. when the clear nature when transparency being good and seeing an image through a film was secured, and the laminating polyethylenenaphthalate film of this invention was excellent in heat ray cut nature, and it was used to the aperture of vehicles, such as an aperture of an automobile, an aperture of an electric car, and an aperture of an airplane, the aperture of a building, etc., having stuck it for a long period of time can be demonstrated, and this invention is very useful.

[Translation done.]

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3. In the drawings, any words are not translated.

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**CLAIMS**

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**[Claim(s)]**

[Claim 1] The compound-ized polyethylenenaphthalate film characterized by having a resin layer in one field of the film with which the laminating of at least three polyethylenenaphthalate layers, an A horizon, a B horizon, and C layer, was carried out one by one, for the resin layer concerned containing a metal addition metal oxide particle or a metal oxide particle, and said B horizon containing a color or a pigment.

[Claim 2] The laminating polyethylenenaphthalate film according to claim 1 characterized by a metal addition gold oxide group particle or a gold oxide group particle being at least one sort chosen from a tin addition indium oxide particle, a stibiation tin oxide particle, an indium oxide particle, and a tin oxide particle.

[Claim 3] The laminating polyethylenenaphthalate film according to claim 1 or 2 characterized by the resin which constitutes a resin layer consisting of activity energy-line hardening resin.

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[Translation done.]